

PATENT SPECIFICATION**(11) 1 504 577****1 504 577**

- (21) Application No. 37633/75 (22) Filed 12 Sept. 1975
 (31) Convention Application No. 2444727
 (32) Filed 19 Sept. 1974 in
 (33) Federal Republic of Germany (DE)
 (44) Complete Specification published 22 March 1978
 (51) INT CL² B22F 1/00 C22C 35/00
 (52) Index at acceptance

C7D 8A2 8A3 8M 8Q 8U 8Z2 8Z5 A1
 C7A 71X A249 A250 A253 A25Y A279 A28X A28Y A300
 A30Y A329 A330 A337 A339 A33Y A340 A341 A343
 A345 A347 A349 A369 A389 A390 A394 A396 A398
 A39Y A400 A402 A404 A406 A409 A40Y A439 A459
 A509 A529 A533 A535 A537 A539 A53X A53Y A541
 A543 A545 A547 A549 A54X A579 A58X A58Y A609
 A629 A671 A673 A675 A677 A679 A67X A681 A683
 A685 A687 A689 A68X A693 A695 A697 A699 A69X
 A70X

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**(54) A PRE-ALLOY POWDER FOR
 THE MANUFACTURE OF ALLOYED
 SINTERED STEEL WORKPIECES**



(71) We, GFE GESELLSCHAFT
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 German Company of 4000 Düsseldorf 1,
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 of Germany do hereby declare the
 invention, for which we pray that a patent
 may be granted to us, and the method by
 which it is to be performed, to be
 particularly described in and by the
 following statement:—

The invention generally relates to a pre-
 alloy powder for the manufacture of
 alloyed sintered steel workpieces, in which
 manufacture a ferro-alloy is first produced
 from the alloy elements required in the
 sintered steel workpieces together with iron
 and carbon, this is pulverised and milled to
 a pre-alloy powder, the pre-alloy powder is
 mixed with ductile iron powder, and the
 mixture is pressed and sintered.

The manufacture of tool steels and high
 speed steels from ferro-alloys is relatively
 new. So far as other steels have been
 manufactured by powder metallurgy, one
 has worked with fully alloyed materials
 manufactured by the melting method and
 subsequently converted by spraying into
 the necessary powder form. These alloy
 powders possess the complete composition
 of the steel that is to be sintered, but have
 the disadvantage of a considerable oxygen
 content. The oxygen content affects the
 mechanical properties of the finished
 workpieces. Moreover very high pressures
 are necessary for pressing, greater by a

factor of 2 than the pressure normal in iron
 powder metallurgy. If one works with lower
 pressures, the tensile strength values
 achieved are insufficient. Steels which
 contain manganese and chromium and/or
 vanadium are not at all practicable to make
 in the manner described. In fact it is very
 difficult to avoid oxidation of the alloyed
 chromium or manganese with the sintering
 atmosphere used in practice. A reduction
 of oxides introduced by these alloying
 elements cannot be accomplished in
 furnaces used for the sintering technique.
 At least if one attempts to work with
 manganese and chromium and/or
 vanadium-additions, the tensile strengths
 achieved by the known process are for this
 reason inadequate.

According to the present invention a pre-
 alloy powder for the manufacture of
 alloyed sintered steel workpieces comprises
 a pulverized ferro-alloy comprising
 manganese and chromium or manganese
 and vanadium in the form of complex
 metallic carbides, with a grain size less than
 10 μm , having an oxygen content of less
 than 0.2% and resistant to oxidation at
 temperatures in the region of 1200°C.

Pressing forces normal in iron powder
 metallurgy, are about 500 MN/m².
 Preferably the sintering temperature is up
 to 1280°C.

Within the scope of the invention
 complex metallic carbides signify carbides

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of at least two of the given elements and iron, mostly in the form of solid solutions.

The invention depends upon the surprising fact that a ferro-alloy consisting of the given complex metallic carbides, when pulverised, does not absorb oxygen, or at least does not do so to a troublesome extent. Therefore the pre-alloy powder, when a protective fluid is used in grinding to a grain-size below $10\text{ }\mu\text{m}$, preferably below $5\text{ }\mu\text{m}$, can without difficulty be limited to an oxygen content below 0.2%, preferably less than 0.15% even if it is milled extremely finely. The pre-alloy powder which is to be used for the manufacture of a sintered workpiece possesses a quite surprising resistance to oxidation, even at temperatures up to 1200°C and more. Difficulties caused by high oxide content of the powder which is to be pressed and sintered to form steel workpieces, and the influence on the tensile strength of the manufactured sintered steel workpieces are eliminated no longer occur. It can be accepted that the complex metallic carbides of manganese plus chromium, or of manganese plus vanadium, effect an additional protection against oxidation. This is also valid with extremely fine milling. It has been found that the alloy elements diffuse very readily during sintering, so that a very homogeneous distribution of the elements is achieved in the finished sintered steel workpiece which has a beneficial effect on the mechanical properties e.g. on tensile strength and hardenability. Examples of ferro-alloy powder and analyses of ferro-alloy powders which are particularly suitable for manufacturing sintered steel workpieces are in % by weight:—

20—25% Manganese
20—25% Chromium
4—8% Carbon
Balance iron

with impurities due to melting

30—35% Manganese
35—45% Chromium
5—7% Carbon
Balance iron

with impurities due to melting.

20—25% Manganese
20—25% Chromium
20—25% Molybdenum
6—8% Carbon
Balance iron

with impurities due to melting.

20—25% Molybdenum
20—25% Vanadium with or without Niobium
20—25% Manganese
Up to 7% Carbon
Balance iron

with impurities due to melting.

In the following is illustrated the use of the pre-alloy powder of the invention in terms of examples of its performance.

EXAMPLE 1

In manufacturing a sintered steel workpiece which contains as alloy elements manganese plus chromium and also molybdenum, normal commercial iron powder is thoroughly mixed with a complex metallic carbide powder according to the invention in proportions of 96% to 4% and pressed as a workpiece e.g. a gearwheel, with a pressure of 500 MN/m^2 . The complex metallic carbide powder contains 21% Cr, 20.8% Mn, 23.1% Mn and 7.8% C, balance Fe.

The milling of the complex metallic carbides took place in an attritor to an FSSS (Fischer Sub-sieve sizes) grain size of $3\text{ }\mu\text{m}$. the oxygen content of the powder amounted to 0.16%. $(\text{Fe, Mn, Cr})_7(\text{C}_3\text{ and } \beta\text{-Mo}_2\text{C})$ were identified in the carbide phases. Sintering took place at 1250°C in a normal sintering furnace, e.g., in a rocker bar heating furnace with a cracked ammonia atmosphere. A test during the sintering process proved that up to 1200°C there was no oxidation of the metallic carbide powder, which, beginning at 1100°C , dissolved easily and completely by 1250°C . in the iron powder.

Example 2

In manufacturing a sintered steel workpiece which contains the alloy elements manganese plus vanadium plus molybdenum, normal commercial iron powder is thoroughly mixed with a complex metallic carbide powder according to the invention in proportions of 97% to 3% and pressed to the desired workpiece with a pressure of 500 MN/m^2 . The complex metal carbide powder contained 21% Mn, 22% Mo and 21.4% V, also 7.9% C balance Fe.

The manufacture of the powder again took place by fine milling in an attritor. The FSSS grain size amounted to $5\text{ }\mu\text{m}$, with an oxygen content of 0.19%. As carbide phases were found: $\text{VC—Mo}_2\text{C}$ solid solutions, and the M_7C_3 type in which M is chiefly iron and manganese.

Sintering took place at 1280°C in a cracked ammonia atmosphere. Again with the complex iron alloy carbide powder used here no oxidation could be conformed up to 1200°C .

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| | WHAT WE CLAIM IS:— | |
| 5 | 1. A pre-alloy powder for the manufacture of alloyed sintered steel workpieces comprising a pulverized ferro-alloy comprising manganese and chromium or manganese and vanadium in the form of complex metallic carbides, with a grain size of less than 10 μm , having an oxygen content of less than 0.2% and resistant to oxidation at temperatures in the region of 1200°C. | |
| 10 | 2. A pre-alloy powder as claimed in Claim 1, wherein the grain size is less than 5 μm . | |
| 15 | 3. A pre-alloy powder as claimed in Claim 1 or Claim 2, wherein the oxygen content is less than 0.15%. | |
| | 4. A pre-alloy powder as claimed in any preceding Claim, consisting of by weight:— | |
| 20 | 20—25% Manganese 20—25% Chromium 4—8% Carbon Balance iron | 5—7% Carbon Balance iron 30 |
| | with impurities due to melting. | |
| 25 | 5. A pre-alloy powder as claimed in any one of Claims 1 to 3 consisting of by weight:— | 6. A pre-alloy powder as claimed in any one of Claims 1 to 3, consisting of by weight:— |
| | 30—35% Manganese 35—45% Chromium | 20—25% Manganese 35 20—25% Chromium 20—25% Molybdenum 6—8% Carbon Balance iron |
| | | with impurities due to melting. 40 |
| | | 7. A pre-alloy powder as claimed in any one of Claims 1 to 3, consisting of by weight:— |
| | | 20—25% Molybdenum 20—25% Vanadium with or without Niobium 45 20—25% Manganese Up to 7% Carbon Balance iron |
| | | with impurities due to melting. 50 |
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